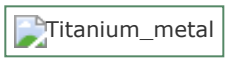
Titanium metal from TiO2 thermite reaction

It's with some pride that I announce the fact that I've produced titanium metal from a homemade potassium chlorate-boosted Anatase thermite...



Earlier, I had developed a sulfur-free potassium chlorate boosted formulation for SiO_2 (silicon) thermites. You can find the rationale for this development on this forum here and on subsequent pages. More on the sulfur-free chlorate boosted formulation for SiO_2 thermite in a separate post soon to come.
[Edit: you can find the post on a sulfur-free silicon thermite here]. And here's a post on Manganese thermite from manganese (II) oxide.

A couple of weeks ago I had adjusted the *best-so-far* $SiO_2/Al/KClO_3/CaF_2$ = 100/96/81/55 formulation to $TiO_2/Al/KClO_3/CaF_2$ = 100/72/61/47 and tried it with my home brewed 'emulsion paint' TiO_2 and it burned but somewhat sputteringly and of course no metal. The home made TiO_2 undoubtedly contained residues of paint resin and possibly other white pigments. Without concentrated sulfuric acid, it is difficult to purify it.

But I recently bought a good grade of pure TiO_2, Anatase "99 %", so I decided to stick that in there too, as well as in an adjusted formulation with magnalium (a 50/50 alloy of aluminium and magnesium - denoted MgAl) . The total charge in both cases was 20 g, contained in an egg cup and embedded in a sand-filled steel bucket. Materials used: very fine Anatase 99 %, 400 mesh Al powder, potassium chlorate reagent grade, ground Fluorite for CaF_2. Ignition with a stoichiometric mix of $Al/KClO_3$ mix and an Mg ribbon fuse. The experimental set-up is very similar to the silicon thermite described here.

The Anatase formulation burns like hell: very fast and furious, very regular: it's completely on a par with its SiO_2 analog. Immediately after the reaction had finished I went over to look and saw three quite large, darker regions at the bottom of the white/yellow hot crucible (eggcup). Darker regions in the hot slag are an indication of materials with higher heat conductivity (such as metals), I've seen this happen many times before.

But at that point I didn't even dare to hope too much and decided to light the magnalium version ($TiO_2/MgAl/KClO_3/CaF_2$ = 100/82/61/49). That basically exploded! I knew it was going to be fast, possibly too fast, so I ran away after lighting the Mg ribbon and the whole thing went "poof!" while I still had my back to it, throwing content as far as a meter away from the test point!

But after the straight Al test had cooled down it became clear that the darker regions were indeed titanium metal: oxidised (only slightly) on the outside on the top and lightly covered in dark slag at the bottom, a little buffing up with rough sanding paper revealed the hard, shiny metal. It has a slight golden tinge to it. I'm now the proud owner of three 4 - 6 mm blobs of home made titanium metal, total weight about 1.9 g.

Interestingly, slag/metal separation was simply exemplary: most of the metal neatly at the bottom, easily separable from the slag mix. It appears to me a lot of the slag had been blown off, covering the globules only in a slight oxide/slag coating. The metal had also passivated, like Al does.

Tonight I'll be running a larger test to confirm.

It may also prove difficult to get chemical confirmation of the metal's identity: Titanium only really dissolves in concentrated acids like sulfuric or nitric, also apparently HF. Nice one... I haven't got any of those...

Update:

Another test using the same formulation but this time with a larger batch size (92 g) also yielded good metal, including one blob of 11.5 g, the thickness of a pound coin but larger in surface. Some smaller globules were spewed out of the crucible, so it's probably running a little *too hot.*

While pure titanium is much more resistant to 32 w% HCl than say steel, it does dissolve slightly, given a bit of time and temperature. The resulting solution is a nice Amethyst type purple, due to aqueous Ti^{3+}. The solution tests positive for Ti with hydrogen peroxide according to this test here. There is therefore no reasonable doubt left that the metal produced is in fact elemental titanium metal.

The reduction of titanium dioxide with Al powder has also been used twice to produce a ferrotitanium alloy (in my case approx. 60 w% Fe and 40 w% Ti) by co-reducing Fe_2O_3 (iron (III) oxide, Hematite) and TiO_2 (here Anatase) with Al powder. The oxide mix was composed of 1 mol of TiO_2 and 0.85 mol of Fe_2O_3. Here, no chlorate-booster is needed because the reduction of Fe_2O_3 to Fe metal provides the heat needed to sustain the reaction and to ensure the reaction products are obtained in molten form. CaF_2 was used as a fluxing agent. Two 20 g reactions were carried out, both yielding exemplary slag/metal separation and clean 5 g reguli of the ferrotitanium alloy.

Without a shimmer of doubt this process can be applied to make other pure titanium alloys of precise composition.

And other heat booster systems besides the potassium chlorate/aluminium powder system have also been tested successfully. A booster system for thermites usually comprises of aluminium and an oxidiser. In the conditions of high temperature that exist inside an ignited thermite mixture, the oxidiser oxidises the aluminium to alumina and the formation of this oxide is accompanied by great release of heat, which is used to keep the thermite 'burning', as well as reach a

sufficiently high end temperature. The latter is necessary to ensure the reaction products, the target metal and slag, are obtained in liquid form and can separate out, thereby obtaining the pure metal as a solid (after cooling).

Other well-known oxidisers for aluminium are nitrates and sulfates. For sodium nitrate the booster reaction is:

$NaNO_3$ + 2 Al ----> Na + 1/2 N_2 + Al_2O_3

This reaction produces about 700 kJ of reaction heat per mol of nitrate.

A typical sulfate is calcium sulfate, used extensively in plaster of Paris, wall fillers, writing chalks and other OTC products and it reacts with aluminium according to:

$CaSO_4$ + 8/3 Al ---> CaS + 4/3 Al_2O_3

It releases nearly 1,300 kJ of heat per mol of sulfate.

I have used both sodium nitrate/aluminium and calcium sulfate/aluminium with success as heat boosters in titanium metal producing thermites (and I've got the metal to prove it!